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Albuquerque Bernalillo County
Water Utility Authority

WATER RECLAMATION DIVISION
4201 2ND STREET SW, ALBUQUERQUE, NEW MEXICO 87105

WATER QUALITY LABORATORY
STANDARD OPERATING PROCEDURE APPROVAL FORM

WQL QA SOP 005
Quality Control/Quality Assurance Procedures

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Quality Assurance Manager

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History of Revision This table lists the revision history and effective dates of this procedure.

Revision	Date	Description of Changes
01		Not Available
02	February 2000	Not Available
03	February 20,2007	2006 A2LA Audit Deficiency-Replaced QA SOP 002 Revised Control Charts Section
04	December 4, 2008	Complete revision, based on input from A2LA Assessor

Water Quality Laboratory
QA SOP 005

1.0 Scope and Application

1.1 This Standard Operating Procedure (SOP) follows the requirements of ISO Standard 17025 and A2LA C205 – SPECIFIC CHECKLIST, Section 5.9.

1.2 The SOP establishes essential procedures for monitoring the validity of tests undertaken and results verification. This standard operating procedure describes the quality assurance/quality control program utilized by WQL, to provide assurance that the quality control duties are being performed effectively.

1.3 To ensure the validity of the analytical data, an established, routine, and rigid quality assurance program is necessary to monitor the reliability (precision and accuracy) of the results reported.

1.4 The use of control charts, see section 2.2 below, allows the laboratory to record data to detect trends in laboratory control samples and assure that necessary corrective actions are taken.

1.5 The objectives of the laboratory quality assurance/quality control program as directed by lab management are:

- ❖ To ensure accountability and traceability of the data.
- ❖ To establish quality control procedures for the determination of acceptable limits of precision and accuracy.
- ❖ To provide rigid guidelines for consistency and strict adherence to standard laboratory procedures adopted by WQL.
- ❖ To ensure that quality control measures are being carried out.

1.6 The basic elements of our QA/QC program are ***control, evaluation*** and ***correction.***

2.0 Responsibilites

2.1 Lab Personnel

- responsible to report all non-conformance of quality control data to immediate supervisor,
- responsible for enteries of data to control charts, and
- responsible to review the control chart(s) for any of the out-of-control situations described in 4.8.

2.2 Lab Supervisors

- responsible for reviewing and validating instrument calibration, standard preparation, method blanks, raw data, calculations and transcriptions.

2.3 Lab Manager

- responsible for ensuring that Standard Operating Procedures faithfully reflect approved published methods, for monitoring trends using control charts updated by the analysts, and for conducting routine internal audits of the laboratory quality system.

2.4 Quality Assurance Manager

- responsibile to plan and organize audits as required by the schedule.
- responsible for routine internal audits and for monitoring the results of quality control check samples during control chart reviews.
- distributes proficiency testing (PT) samples, which determinations assure quality of test results.
- monitors annual method detection limit (MDL)

3.0 Internal Audits

3.1 Audit Requirements

3.1.1 Internal audits are conducted annually and a predetermined schedule is posted. The audit verifies that WQL operations comply with the requirements of the management system, A2LA requirements, and ISO/IEC Standard 17025.

3.1.2 Auditors must be trained and qualified personnel who are independent of the activity to be audited. Training records must be on file with personnel records in QA file room. These records must demonsrate that the internal auditors are trained and qualified.

3.2 Procedures for Conducting Internal Audits

The following procedures are applied when conducting internal audits at WQL:

3.2.1 Applicable procedures will be audited a minimum of once annually or at any time lab management feels that an audit is necessary or results are questionable an unscheduled audit may be performed.

3.2.2 The audit schedule will be determined and posted every July. All internal audits must be completed before the end of December.

3.2.3 The specified areas to be audited include Quality Assurance, Sample Receiving, Chemistry, Nutrients, Demands, Microbiology, Metals, purchasing, client complaints, nonconforming work, corrective actions, past internal audits, action plans, and management reviews.

3.2.4 A2LA checklists or WQL approved checklists, when available, will be used during the audits. The minimum review and validation requirements for internal audits are as follows:

- Verification of implementation of written policies and compliance with

written procedures.

- Verification and documentation of procedures for confidentiality.
- Observation of standard operating procedures, evaluation of the procedure for adequacy and precision, and evaluation of correct safety procedures.
- Confirmation that required quality control procedures are followed for each analytical method.
- Review of sample documents for completeness by the analyst(s) at each step of the analysis.
- Review of instrument logs, performance test results, and analyst performance.
- Review of analytical data and random calculation checks.
- Review of purchasing supplies that may affect the test method.
- Review of all complaints pertaining to test method audited.

3.2.5 Prior to performing an audit, concerns raised during the previous audit and/or management reviews must be addressed. This will allow the auditor the opportunity to verify correction of previous deficiencies and compliance with corrective actions.

3.2.6 Corrective actions in response to audit deficiency findings are due to the assigned auditor 30 days after completion of the audit. Auditors may provide a summary report of all deficiencies, however, CARR procedure will be implemented for all deficiencies (See QA SOP-003).

3.2.7 After the auditor reviews the corrective actions all deficiencies will be presented to WQL personnel during a QA training meeting.

4.0 Control Charts

4.1 Theory of Control Charts

The performance of a measurement system can best be demonstrated by the measurement of a stable and homogeneous control sample in a planned repetitive process. The data generated is plotted as a control chart to indicate whether the measurement is statistically in control. The control chart warns the laboratory of possible deviation from 95% confidence level by identifying errors, drifts, or other types of assignable variations.

4.2 Uses of Control Charts

- Provide a graphical representation of accuracy and precision for the analysis of each analyte and instant detection of erroneous data.
- Allow efficient observation of control trends for a particular analysis and provide long-term mechanism for self-evaluation of analytical data.
- Provide assessment of analytical capability of the analyst with regard to the output of valid analytical data.

- Allow observation of deviations from control trends.

4.3 Types of Control Charts

- Percent Recovery – verification of accuracy
- Relative Percent Difference – verification of precision

4.4 Batch Requirement

Analysis of each batch of samples include a LCS, a LCSD, and a Laboratory Reagent Blank (LRB) within a batch of twenty samples or less, as appropriate to the test method. These quality control samples are taken through all steps of the method, including preparation and analysis. The LCS sample is used for measuring the precision (percentage recovery) of the method, while the LCSD result is compared against the LCS result to measure the accuracy (RPD).

Within the control chart system, which is set up in the lab share network drive, four specific measurements are evaluated: LCS-percent recovery, LCS-percent difference from the true value, LCSD-% difference from the measured LCS, and the LRB result compared to the MDL (control limit is 2.2 times the MDL). Warning limits are measured, statistical trends are observed, analyst performances are evaluated, and analyst inputs are considered.

4.5 Control Chart Calculations

The mean and standard deviation of percent recovery (%R) are calculated using LCS and LCSD results entered into the Excel control chart spreadsheet. Relative percent difference is defined as the difference between two sample results divided by their mean and expressed as a percentage.

From this data, the upper and lower control limits and warning limits are calculated.

Upper Control Limit (UCL) = Mean of %R + 3s
Lower Control Limit (LCL) = Mean of (%R) - 3s where *s* denotes standard deviation and is defined as the square root of the average of the squares of deviations about the mean of a set of data.
The (%R) of each Laboratory Control sample is plotted on a control chart and compared with statistically generated control limits.

Warning Limit (WL) = Mean of (%R) +/- 2s

Data precision is evaluated on the results of the samples analyzed in duplicate. The range is calculated and divided by the average of the two analyses, then multiplied by 100. This value equals the relative percent difference (RPD). The results of RPD are plotted and the resultant graph evaluated in terms of deviation from historical values.

4.6 Estimating Uncertainty

An estimated uncertainty is required for every analytical result reported by WQL, as part

of ISO/IEC 17025. The definition of the term uncertainty (of measurement) is, "A parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand."

The measurement uncertainty is calculated using the standard deviation from a significant number of LCS results. The standard uncertainty is defined as the standard deviation of the LCS measurements divided by the square root of the number of measurements. This value is automatically calculated on the control chart spreadsheet. It is recommended that a significant number, a minimum of six months of data, of individual LCS data points be obtained to calculate measurement uncertainty in order to represent an appropriate expanded uncertainty using the following calculation:

Measurement Uncertainty for a Defined Matrix (LCS) = 95% Confidence Level = 2 x sd

4.7 Procedures for Data Entry into Control Charts

1. Laboratory analyst enter control chart data daily in the J lab share drive as per assigned tasks.
2. Each analyst enters the control chart data into the applicable control chart spreadsheet and reviews the control chart for any of the out-of-control situations described in 4.8.
3. If the quality control data is not within limits, notify immediate supervisor and/or initiate CARR procedures (See QA SOP-003).
4. Once the supervisor has signed off on the reported results, the analyst may proceed with data entry into SQL*LIMS.
5. If quality control data is within limits, the control values for LCS and LSCD are plotted on the control charts.
6. Each control chart is set up by parameter with at least 25 points on the graph at all times.
7. The analysts must also evaluate UCL, LCL, and Warning Limits. The set limit for each parameter is set at 85-115% Recovery and for any limits that do not meet this criteria a CARR must be initiated and must following QASOP003 procedures.

4.8 Procedures for Evaluating Control Charts

1. Control chart reviews will be conducted weekly. The objective of the control chart review is to identify components of uncertainty and bias data, while making a reasonable estimation of uncertainty, and ensuring that reported results are giving a reasonable impression of accuracy.
2. The objectives of the analyst and results reviewers' control chart reviews are to assure data quality prior to entry into SQL*LIMS.
3. Control charts will be evaluated at a minimum of twice per year per control chart. It is best to have at least 25 data points in each chart. If there are less than 25 data points available, then use what data is available. Control charts are updated by the QA section from one review session to the next for the particular parameter being evaluated.
4. Reviews will include verification of logbook entries in correspondence to entries

on Excel control chart spreadsheets, control of the control chart spreadsheets and calculations within the quality system, and the effectiveness of any corrective actions taken on control charts.

5. Reviews will also include evaluating criteria set forth in section 4.9
6. Prior to reviews the QA section will review the chart for accuracy and to verify the uncertainity values and the average baseline.
7. Lab personnel will be assigned the task to review control charts on a weekly basis. A Control Chart Review form will be filed with QA.

4.9 Criteria for Evaluating Control Charts

1. In the reviews lab personnel will look for trends in the existing data. The list of criteria below is the formal definition of a "trend."
2. Each one of the following criteria will be evaluated in the reviews and documented on the Control Chart Review form for each analyte.
3. This criteria will be used to evaluate both % Recovery and % Difference control charts.
4. The lab personnel must check the following for each analyte to determine if the analyses conducted are in statistical control:
   - Number of individual points above the Upper Control Limit.
   - Number of individual points below the Lower Control Limit.
   - Seven points in a row all above average or all below average for % Recovery or average RPD for % Difference.
   - Seven points in a row increasing.
   - Seven points in a row decreasing.
   - Ten out of eleven points in a row all above average or all below average or average RPD.
   - Cycles or other non-random patterns in the data.
   - Two out of three points in a row outside of two standard deviations above the average, or two out of three points in a row outside of two standard deviations below the average.
   - Four out of five points in a row outside of one standard deviation above the average, or four out of five points in a row outside of one standard deviation below the average.

4.10 Out-of Control Data on Control Charts

A control chart may indicate an out-of-control condition either when one or more points fall beyond the control limits, or when the plotted points exhibit some nonrandom pattern of behavior.

The process is out of control if any one or more of the criteria is met.

1.One or more points outside of the control limits. This pattern may indicate:
   - A special cause of variance from a material, equipment, method, or measurement system change.
   - Error in measurement of a part or parts.
   - Miscalculated or misplotted data points.

- Miscalculated or misplotted control limits.

2. Four of five consecutive points beyond the control limits. This pattern indicates a shift in the process output from changes in the equipment, methods, or materials or a shift in the measurement system.
3. Two of three consecutive points outside the warning limits but still inside the control limits. This may be the result of a large shift in the process, in the equipment, methods, materials, or operator or a shift in the measurement system.
4. An unusual or nonrandom pattern in the data.
    1. A trend of seven points in a row upward or downward. This may show:
        - Gradual deterioration or wear in equipment.
        - Improvement or deterioration in technique.
    2. Cycling of data can indicate
        - Temperature or other recurring changes in the environment.
        - Differences between operators or operator techniques.
        - Regular rotation of machines.
        - Differences in measuring or testing devices that are being used in order.
5. Several points near a warning or control limit.

4.11 Procedures for Reporting Out-of Control Data of Control Charts

If any of the criteria of 4.10 exists, the cause of out of control data points should be determined and verified that each was a special case that will probably not recur. If the condition is reoccurring then a Corrective Action Response Report (CARR) report will be initiated-See QA SOP-003 for specific instructions.

4.12 Annual Evaluation of Control Charts

As part of the Annual Management Review, the lab management will evaluate the data collected from the control chart reviews.

The requirements for annual control chart reviews are:

- The laboratory's statistical control limits that will be used as guidelines to validate the data generated.
- Recommendations for any changes to the control limits for the following year.
- Review of performance indicators such as blanks, LCS recoveries, LCSD percent differences.
- Review of analyst performance and suggestion for improvement of procedures.
- Consistent entry of data points.
- Total points outside warning limits.
- Trends or shifts in deviation and incidences of out of control data.
- Non-random patterns or trends in the data.
- Changes in uncertainty estimate.
- Effectiveness of corrective actions taken during the past year.

In addition, the uncertainty, and warning and out-of-control limits are compared to previous reviews to evaluate any significant variations that may have occurred from one review to the next. Uncertainty sources are identified, which may include, but are not limited to the following areas:

- Storage Conditions
- Instrument effects
- Reagent purity
- Measurement conditions
- Sample effects
- Computational effects
- Blank Corrections
- Operator effects
- Random effects

5.0 Proficiency Testing

5.1 External Proficiency Testing (PT)

In order to verify that WQL possesses the capability to provide accurate and reliable test data in its day-to-day operations and to maintain high standards of performance, a competent, disinterested third party is necessary to evaluate the laboratory's performance based on personnel, physical facility, instrumentation, and quality assurance/quality control programs. Analysts proficient in the procedures involved perform two Water Pollution (WP) studies, two Water Supply (WS) studies and one Discharge Monitoring Report Quality Assurance (DMR-QA) study annually as assigned by laboratory management.

5.2 Four Year PT Plan

- DMR-QA, WP and WS studies will be performed during June of each year from 2008 to 2012.
- WS and a WP study will be performed during October of each year from 2008 to 2012.
- A contract with Environmental Resource Associates (ERA) exists for provision of the samples for these studies. The current contract expires March 4, 2009 with two one-year extensions available.

5.3 Procedures for Analysis of PT Samples

It is essential that proficiency testing samples are treated as routine samples. Analyses of proficiency samples should not be repeated, unless it is necessary to repeat the entire procedure and samples should be run in duplicate *only* in those procedures where samples are normally analyzed in duplicate. In addition, only lab analysts with current DOC's (See QA SOP-004) are allowed to analyze eternal PT samples. Eternal PT samples will not be used for training purposes.

Supervisors are individually responsible to ensure that the performance and evaluation of proficiency samples are submitted within the designated time frame. The Laboratory Manager reviews all data and forwards the packet to the QA Manager by the date defined when issued.

5.4 Submission of Results to A2LA

Results are entered in the eDATA website of ERA. Copies are retained in the QA file room. The results are reviewed upon receipt of the summary report. Copies of the summary report are distributed to lab management by the QA Manager. WQL performance for analysis of PT samples is evaluated and reported to A2LA, State of New Mexico, and EPA Region 6 by ERA.

5.5 PT Corrective Action Process

A CARR is initiated according to WQL's corrective action procedures for all "unacceptable" results. All unacceptable results are investigated by the Lab Manager and the cause or causes identified for the unacceptable performance, and corrective action implemented.

Completed CARR and/or Corrective Action Summary Reports are submitted to ERA within 30 days of receiving the summary report. It is the responsibility of the Lab Manager to complete all CARR's for unacceptable results. It is the responsibility of the QA Manager to submit all completed CARR's to ERA.

5.6 Internal Proficiency Evaluation (PE)

In order to comply with the WQL training program, analysts are issued in-house PT samples as part of the WQL Demonstration of Capability process (See QA SOP 004). In-house PT samples are provided to analysts by the QA section, which evaluates all in-house proficiency evaluation samples. Complete records are on file in QA file room.

6.0 Method Detection Limits (MDL)

EPA defines MDL as "the minimum concentration that can be determined with 99% confidence that the true concentration is greater than zero." This procedure is outlined in 40 CFR 136, Appendix B. WQL's MDL form outlines these requirements and must be followed when determining method detection limits.

6.1 Procedures for Analysis of MDL's

1. MDL's are determined **annually** or whenever, in the judgment of the lab management, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate that the MDL must be re-determined.
2. Make an estimate of the detection limit using on of the following procedures:
   - Concentration value corresponding to instrument signal/noise in the range of 2.5-5.
   - Concentration equivalent of 3 times the standard deviation of replicate instrumental measurements of the analyte in reagent water.

- The region of the standard curve where there is a significant change in sensitivity, i.e., a break in the standard curve.
- Instrument limitations.

3. Make a standard 1-5 times the estimated detection limit.
4. Seven replicate aliquots of the standard are processed through the entire analytical method.
5. All calculations defined in the method are completed and the concentration values are reported in the appropriate units.
6. MDL is calculated as follows:

   MDL = (t) x (S)

   where: t = students' t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom {t=3.14 for seven replicates].

   S = Standard deviation of the replicate analyses.
7. If additional confirmation is desired, the seven replicate aliquots are reanalyzed on two or more nonconsecutive days and MDL values are recalculated for each day. An average of the three MDL values for each analyte may provide for a more appropriate MDL estimate.

6.2 Reporting MDL's

1. Lab analysts are responsible for turning in a MDL Development Form for each analyte that is run to their immediate supervisors.
2. Supervisors must validate the analysis, initial the form and forward to Lab Manager.
3. The Lab Manager will review the information and forward to the QA staff.
4. The QA staff will record the MDL value in the J lab share drive and will make the corrections in SQL-LIMS, if required. The QA staff will report all corrections in LIMS to the Technical Program Manager.
5. The Technical Program Manager will notify clients when MDL's have been changed.

6.3 Use of MDL's

1. The MDL's will be used to set reporting limits in SQL-LIMS. The method detection limit is <u>not</u> the lowest limit WQL can accurately test during routine analyses – the lowest limit is actually at least 2.5 times higher than the MDL. Therefore, the reporting limit is the MDL times 2.5 to 5, to account for matrix effects in client samples. The actual multiplier is determined based on best professional judgment of laboratory staff.
2. The MDL may also be used to determine an amount to "spike" a sample for a known addition. Generally, samples are spiked at 5 to 50 times the MDL.
3. The MDL annual reviews may be used to detect problems with procedures. If the MDL is much higher than the estimated detection limit, or if over the course of time the MDL steadily increases, it may indicate problems with the procedure or with the analytical equipment. Some things to check for may include improper lamp alignment, low lamp output efficiency, expiration dates for chemicals,

improperly made reagents, etc. If a problem is detected a CARR must be initiated and QA SOP-003 be implemented.

7.0 Data Assessment

Data assessment incorporates a variety of techniques to evaluate the quality of the measurement process and the data generated. Of major importance are precision determination, accuracy evaluation, data validation, data verification and data reporting.

7.1 Precision

Analytical precision, Relative Percent Difference (RPD), is expressed as a percentage of the difference between the results of laboratory control samples, and calculated as follows:

LCSD Calculation: $\frac{[LCS-LCSD]x\ 2}{LCS+LCSD}$ X 100 (%Difference in lab control duplicates)

MSD Calculation: $\frac{[MS-MSD]x\ 2}{MS+MSD}$ x 100 (%Difference in matrix spike samples)

7.2 Accuracy

The accuracy of measured data is evaluated by the comparison of the percent recovery of the matrix spiked sample analysis. Statistically based control limits have been established for the LCS for each method of analysis and sample matrix. Recoveries are assessed to determine method efficiency and matrix interference effects. Analytical accuracy is expressed as the percent recovery of an analyte/parameter, which has been added to the samples ("spiked") at a known concentration before preparation and analysis. The equation used to calculate percent recovery (%R) is as follows:

MS Calculation: $\frac{Cs-C}{Cs}$ x 100 (%Recovery of matrix spike)

LCS Calculation: $\frac{LCSs-LCS}{LCSs}$ x 100 (%Recovery of lab control standard)

In the formulas above, Cs means spiked matrix concentration, C means measured matrix concentration, LCSs means spiked reagent water concentration, and LCS means measured reagent water concentration.

7.3. Data Review and Validation

For data to be valid, it must meet all the acceptance criteria, including accuracy, precision, and any other criteria specified by the analytical method used. Data review and validation procedures are employed to ensure that the reported data are free from transcription and calculations errors. It is the responsibility of laboratory personnel to follow these procedures

to ensure that all quality control measures are reviewed and evaluated before the data is reported. The following is the level and responsibility for validation procedures.

Analysts Level (Daily Data Collection)

- Check all QC data for acceptability
- Enter QC data into control chart
- Review control chart for acceptability
- Check all data for transcription errors
- Check manual calculations
- Correct all errors using a single line cross-out, initial, and date.

Note: Identify all unreadable results (write overs, cross-outs, or unreadable results) in a correction on the logsheet. If the data are not available to verify the correct data, initiate a CARR.

Reviewer Level (Daily Data Collection)

Note: Data reviewers must be an analyst with a current DOC in the analysis to be reviewed or a member of the WQL Management Staff.

- Review all data recording for transcription errors write overs, cross-outs and legibility.

Note: Identify all unreadable results (write overs, cross-outs, or unreadable results) in a correction on the logsheet. If the data are not available to verify the correct data, initiate a CARR.

- Review all QC data for acceptability (see Section 4.0)
- Review control chart for acceptability (see Section 2.2.5)
- Check all data for transcription errors
- Check manual calculations
- Sign and date the data review section on the logsheet.

Note: Data are now ready for entry into SQL*LIMS.

Supervisor Level

- LIMS Sample Level Validation
  - Check suspect entries
  - Approve completed samples that are SUSPECT/NOT APPROVED/COMPLETE ONLINE
- 5% Daily Report: Validate randomly selected results fully through prep/analysis/data entry
  - Print out report from SQL*LIMS
  - Check all data on form for the items listed above for data reviewer.
  - Stamp and sign the reviewer information onto the form.
  - Scan completed form and e-mail to Laboratory Manager.
  - Provide original form to QA Manager.

Note; Initiate CARR for any errors detected in the 5% validation.

- QA/QC out of spec
  - Supervisor signature required for all out of specification data {excluding MS/MSD}

- Confirm QC samples to assure the accuracy of the test method including calibration and/or continuing calibrations and use of certified reference materials.
- Selection and use of reagents and standards of appropriate quality

Manager Level

- LIMS Submission Level Validation
  - All SUSPECT samples: Review and manually approve submission
- Repeated QC out of specification
  - Initiate Corrective Action Response Report
  - Customer Liaison: Inform Technical Program Manager of potential suspect results and corrective actions planned.
- Assure the selectivity of the test for its intended purpose
- Annual Control Chart Reviews

7. 4 Computer Generated Calculations

For those spreadsheets that are used for calculation purposes in analytical procedures, a quarterly verification of these spreadsheets must be conducted. Records are filed with QA section.

7.5 Data Reporting

All laboratory data is entered via SQLLIMS system. Laboratory analysts are responsible for entry of the results they produce each day.

7.6 Placing Text on the Sample

Text comments may be placed on template and instance records in SQLLIMS. This activity is encouraged in the laboratory and constitutes a vital form of documentation and communication regarding the status and condition of samples and tests associated with the sample. In SQLLIMS text is allowed at all levels of an instance record. Currently, WQL only uses the Sample Text record for comments. The text record must begin with the log-in initials of the person providing the comments and the date in DD-MON-YY format and a brief description of what happened or conditions discovered. Any unusual appearance, odor, or observation such as floating material, foam, sediment, in a sample container is reason for text. Any other characteristic atypical for the type of material submitted is justification for text.

8.0 Assuring the Quality of Test Results

All quality control measures shall be assessed and evaluated on an on-going basis, and quality control acceptance criteria must be used to determine the usability of the data. The minimum requirements of this QC program consist of an initial demonstration of capability (refer to QA SOP-004), and periodic analysis of laboratory reagent blanks, fortified blanks and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data thus generated.

8.1 Quality Control Requirements

- **Calibration**

*Calibration Curve - Blank & 3 Standards
*Calibration Verification - Blank & Standard

- **Sample Analysis**
  *Laboratory Reagent Blank [LRB]- < MDL
  *Laboratory Control Sample and Duplicate [LCS/LCSD]-Minimum one each per batch
  *Laboratory Matrix Sample and Duplicate [MS/MSD] - Minimum one each per batch

- **Periodic Requirements**
  *PT Samples – As scheduled
  *Method Detection Limit - Annually
  *In-house PT Samples – As required for DOC certification

8.2 Quality Control Limits
Assurance of quality analytical results is achieved by applying the following control limits to all analytical methods.

8.2.1 Quality Control-Laboratory (Method) Performance
In addition to the necessary calibration standards, blanks, duplicates, spikes, reference materials and/or control samples are to be included at the rate of one in twenty samples or one of each set of runs of less than twenty samples (See QA SOP007 for definition of Batch).

MS – Matrix Spike – measure the effects the sample matrix may have on the analytical method, usually the analyte recovery. Method accuracy is documented and controlled based on the percent recovery of matrix spikes for quantitative analysis and the positive response of the analyte for qualitative analysis.

MSD- Matrix Spike Duplicate - measure precision of the analytical process. Method precision is documented and controlled based on the relative percent difference (RPD) or the positive response for qualitative analysis.

LCS - Laboratory Control Sample - measure method performance. The matrix of the LCS should match the matrix of the samples being analyzed and should pass through the entire sample preparation process. The LCS, therefore, measures both the sample preparation process and the analytical process.

LCSD -Laboratory Control Sample Dup - measure precision of the analytical process. Method precision is documented and controlled based on the relative percent difference (RPD) or the positive response for qualitative analysis.

LRB - Laboratory Reagent Blank- either matrix or reagent, determine and measure contamination and interferences - The results of blanks should be compared with the sample analyzed per analysis to determine whether the source of any analyte present is due to sample or laboratory contamination, interferences, the sample matrix, or the actual

analyte in the samples. Blanks should be below the method detection limit where possible. Blank results are evaluated and corrected where possible. If blank results are consistently above the method detection level (MDL) established, the MDL should be re-established. High blank results may also indicate contamination either from the solvent, laboratory equipment or laboratory environment.

8.2.2 Quality Control-Instrument Performance

Inclusion of standards in analytical methods is necessary to quantify the concentration of analytes present in the sample. It establishes a reproducibly reference point to which all sample measurements can be correlated. Calibration is the process for determining the correctness relative to physical or chemical standards. A set of initial calibration standards (ICAL) and a secondary source initial calibration verification standard (ICVS) are analyzed before sample testing begins to verify calibration. In addition, a continuing calibration verification standard (CCVS) is analyzed after every 10 samples and at the end of sample batch. This standard is necessary to verify the standard curve and may serve in some cases as sufficient for calibration.

ICAL - Initial Calibration Standards - Calibration curves are plots of the instrument response versus concentration. Typically, the plot will be linear. A plot is defined as linear if the correlation coefficient (R) calculated from linear regression analysis is 0.995 or greater. Each component being analyzed is standardized by analysis of at least three standards and a blank. The standard concentrations should be evenly distributed throughout the range of the method.

ICVS - Initial Calibration Verification Standard/ CCVS- Continuing Calibration Verification Standard - Calibration check standards referred to as initial calibration verification (ICVS) and continuing calibration verification (CCVS) are used to determine whether an analytical procedure is in control and stays within control. They are used to detect analytical method errors from procedural or operator errors or contamination from laboratory sources.

8.3 Quality Control Checks and Corrective Actions

8.3.1 Out-of –control data

Whenever the analytical process is out-of-control, one or more of the following individuals will initiate investigation/corrective action:

- The **analyst** must be able to recognize out-of-control conditions and immediately notify the Laboratory Supervisor for action plan.
- The **Laboratory Supervisor** must review analytical and Quality Control data for reasonableness, accuracy, calculation errors, and completeness.
- In the event of an out-of-control analysis, the Lab Supervisor works with the analyst to solve the problem and prevent the reporting of suspect data by stopping the work in question and ensuring that all results that are suspect are repeated, if possible, after the source of the error is remedied. If the problem is

a continuing problem, a Corrective Action Response Report (CARR) must be initiated (see QA SOP 003 for Corrective Actions and Preventive Actions procedures).

- The Laboratory Operations Manager must evaluate the causes of a continuing out-of-control analysis documented in a CARR and report the findings to the Technical Program Manager and Quality Assurance Manager.

8.3.2 Acceptance Criteria and Corrective Actions
See Attachment

9.0 Ethics and Data Integrity

9.1 Policy
WQL is committed to reporting only data, test results and conclusions that are accurate, precise and of the highest quality. WQL has developed this Ethics and Data Integrity Policy that must be adhered to by all laboratory personnel.

Under the policy, WQL employees are prohibited from:

- Altering an instrument computer or clock for any inappropriate purpose;
- Altering the contents of logbooks and/ or data sheets to misrepresent data;
- Misrepresenting an analyst's identity;
- Changing raw data documents without approval;
- Inappropriate calibration techniques such as peak shaving, setting fraudulent integrator parameter, use of computer macros that alter QC results, or altering testing controls;
- Changing reported results without proper documentation and approval;
- Altering injection volumes for calibration and misrepresenting the true values;
- Failure to comply with standard operating procedures or methods without proper documentation and approval;
- Any attempt to misrepresent data or events as they actual occur in the course of data production, review, or reporting;
- Disposing of or deleting electronic data files or hardcopy of raw data;
- Engaging in any practice that ultimately misrepresents data or narratives in any way.

WQL employees further will not tolerate unethical practices by others and are instructed to inform the Laboratory Management of any accidental reporting of non-authentic data within 24 hours of discovery.

An employees who is aware of misrepresentation of facts regarding analytical data, or any manipulation data, are required to immediately inform his/her supervisor, who, in turn, must notify the Laboratory Management.

Each employee is required to attend a training session on ethics and data integrity and sign an Ethics and Data Integrity Agreement affirming that they understand the policy. Employees are also required to attend annual refresher sessions.

9.2 Training Program Procedures

Ethics and data integrity training includes the following steps:

1. The QA Manager reviews the policy with all lab personnel as a group or individually for new hires. The importance of the policy is discussed and each employee is given a copy of the Data Integrity Policy. The reviews will be conducted before January 31 of each calendar year.
2. The topics identified in the policy statement above are discussed .
3. Employees are encouraged to participate in-group discussions identifying other topics that may be added to the above list or may give examples of what they would consider misrepresentation of analytical data.
4. After group discussions, the QA Manager encourages lab personnel to meet individually with the QA Manager and/or Technical Program Manager if there are further questions. If there are no further questions, the employee signs and dates the Ethics and Data Integrity Agreement.
5. The training record and a copy of the Agreement are stored in employee files and are reviewed every January. The second page of the agreement includes signatures and dates for recording renewals.
6. For new hires, the policy is reviewed prior to performance of the first Demonstration of Capability test.

9.3 Audit of the Ethics and Data Integrity Program

- On an annual basis the QA Manager monitors ethics and data integrity during internal audits.
- During the internal audit process the following is audited as part of data integrity:
  - Laboratory Control Samples/Laboratory Control Samples Duplicates QC data- for all results that are reported outside the limit an associated CARR with a client notification must be presented during the audit. For results reported outside the limit, without a CARR or client notification, a deficiency will be written as a separate internal audit report.
  - Emails, phone calls, or other client notifications may be audited to ensure the data integrity policy is being implemented.
  - Employees, including supervisors and managers, are interviewed during internal audits to ensure standards of integrity are being followed.
  - If the policy has been violated a Corrective Action Response Report is initiated and following a full investigation..

Quality Control Checks and Corrective Actions for the Laboratory			
QC Activity	**Frequency**	**Acceptance Criteria**	**Recommended Corrective Action**
ICAL'S - Instrument Calibration	Prior to analysis and if continuing verfication CCVS not met	Correlation coefficient equal 0.995or >	Re-analyze standards. If same response is obtained, re-calibrate instrument and re-start analysis. If same response is obtained, prepare new standards and re-start analysis.
ICVS - Initial Calibration Verification	One per each calibration curve produced	-Second Source Vendor or SOP %10± Generally specific	Re-analyze standards. If same response is obtained, re-calibrate instrument and re-start analysis. If same response is obtained, prepare new standards and re-start analysis.
CCVS - Continuing Calibration Standards	Initial + every 10th Sample + end of each run	Within 5% for Metals ±10% for Chemistry or SOP specific	Recalibrate and re-analyze the affected portion of the analysis. Accepted portions of the analysis must be bracketed by acceptable QC checks.
MS- Matrix Spike Samples	One per batch of 20 samples or less	85 - 115% recovery or SOP specific. DO NOT average MS and MSD results	Source of the problem should be identified and samples must be qualified by texted in SQL-LIMS for matrix interferences.
MSD- Matrix Spike Duplicate	One per batch of 20 samples or less	85 - 115% recovery	Source of the problem should be identified and samples must be qualified by texted in SQL-LIMS for matrix interferences.
LCS - Labortory Control Sample	One per batch of 20 samples or less	85 - 115% recovery at the same spiking level as the matrix spike	Re-prep batch and re-analyze. If acceptable, re-analyze affected portions of the analysis. If not acceptable, check for spiking solution degradation or contamination, dispenser/pipette calibration, or instrument calibration problems. Initiate for CARR procedures for all non-conformance.
LCSD - Laboratory Control Sample Duplicate	One per batch of 20 samples or less	±10% Difference	Source of problem should be identified and resolved. Initiate CARR procedures for all non-conformance.
LRB and Cal Blanks -Lab blanks (method, reagent, digestion, instrument)	One per batch of 20 samples or less. For instrument use need to analysis with each calibration and prior to CCVS's	< or equal to 2.2X MDL	Prepare new blank and re-start analysis. If same response is obtained, determine cause of contamination (reagents, calibration standards, environment, equipment failure, etc.) and minimize or eliminate. If different response is obtained, re-analyze samples if possibility of being affected by the initial contamination problem exists. Flag associated data if the concentration of a targeted analyte in the blank is at or above the reporting limit and is greater than 1/10th of the amount measured in any sample; or affects sample results per the test method requirements.